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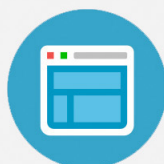J. Vac. Sci. Technol. B **17**, 731 (1999); 10.1116/1.590629

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Enhancement in field emission of silicon microtips by bias-assisted carburization

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Ultrathin carbon layers with thicknesses below 50 Å have been deposited on silicon microtip arrays by bias-assisted carburization (BAC) using microwave plasma chemical vapor deposition. The tip radius of these silicon tips is reduced below 55 nm under low deposition temperature. The field emission characterization has been performed in a high-vacuum environment. An enhancement in the field emission is observed of about 3 orders of magnitude in BAC silicon microtips over untreated silicon microtips. With an applied voltage of 1100 V, emission currents of 80 and 120 μA have been achieved for the films grown (at dc bias of -200 V for 40 min) with 15% and 25% CH_4/H_2 gas ratio, respectively. An emission current of 40 μA has been achieved for the film grown (at dc bias of -300 V for 40 min) with 3.5% CH_4/H_2 ratio. The BAC silicon emitter has good emission stability at a constant voltage of 1100 V. These investigations indicate that further improvement of this technology will lead to simple and inexpensive field emission display devices. © *2000 American Vacuum Society.* [S0734-211X(00)10706-1]

I. INTRODUCTION

Field emitter arrays are attracting a great deal of attention as a promising electron source for field emission display devices.[1,2] Hence, there is a considerable surge in research activity to find a superior material for field emitters. Among the various materials, the use of sharp silicon microtips is attractive for field emitter arrays. However, the progress in such sharp silicon microtips is hindered by its high work function, low electron conductivity, and poor stability. To enhance its emission efficiency and stability, various coatings on the surface of silicon microtips have been reported.[3–5] In recent years, diamond has been considered a promising emitter material for field emission devices that exhibits several unique properties such as a negative, or at least a very small, electron affinity of {111} or {100} oriented facets, high thermal conductivity, and outstanding chemical and physical stability properties.[6–10] However, it is difficult to fabricate a conical diamond emitter due to its high hardness and chemical inertness. Moreover, conventional chemical vapor deposition (CVD)-grown diamond requires high temperature for deposition, which is incompatible with most silicon processes.

Another potential material is carbon nanotubes, which have been recognized as an important nanoscopic system for field emitter applications;[11–15] but it still remains unclear how to scale up the novel devices based on carbon nanotubes because of the lack of scale-up growth strategies and large-scale nanotube assembly strategies.[16] It is also reported that *a*-C:H has a low electron affinity[17] and exhibits very good cold-cathode electron emission characteristics.[18] It is therefore worthwhile to deposit amorphous carbon film on silicon microtips by a simple and easy method and to enhance the emission performance of sharp silicon microtips as their fabrication process is somewhat more established.

In this article, systematic studies have been performed by the field emission from silicon microtips arrays covered with and without ultrathin carbon layers deposited by bias-assisted carburization (BAC) using a microwave plasma CVD technique with various CH_4/H_2 gas ratios, substrate dc bias, and duration of bias. These ultrathin carbon layers on silicon microtips are further characterized by scanning electron microscopy (SEM) and Auger electron spectroscopy (AES). The field emission characteristics of thin BAC-treated silicon microtips as compared to bare silicon have been investigated. Comparison of the field emission properties and morphologies of these silicon microtips leads to an easy way to control the process parameters such that it does

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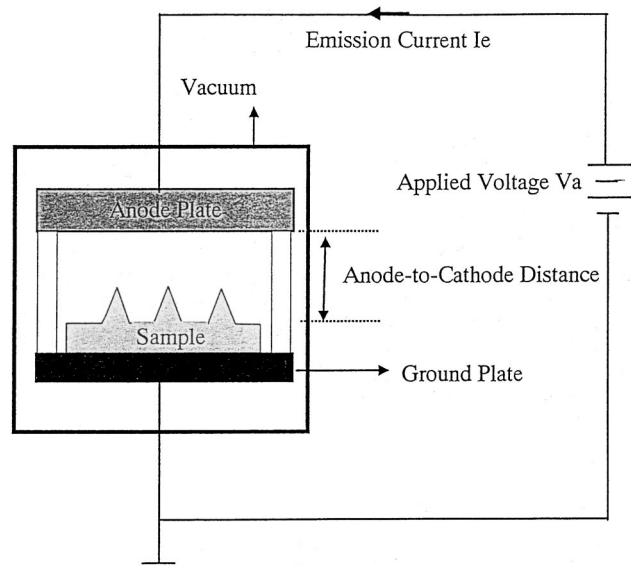
not affect the sharp apex, but only improves the surface properties for better electron emission.

II. EXPERIMENT

A. Fabrication of BAC-silicon monotips

An oxide layer 1 μm thick was thermally grown and photolithographically patterned to form arrays of 50 $\times$ 50 squares with 1 μm in width on an *n*-type silicon substrate. The patterns of photoresist were then transferred onto the underlying oxide using dry etching. With the prepatterned oxide as the mask, high-aspect-ratio cone-shaped silicon microtip arrays were subsequently formed using SF_6/Cl_2 reactive ion etching. These etched tips were further sharpened by oxidation and a stripping process[19] and were then cleaned by hydrogen plasma. Silicon microtips prepared by this procedure are used to deposit a carbon layer with a thickness below 50 Å by the BAC process.

B. BAC on silicon microtips

The BAC process was performed using an AsTeX microwave reactor. The carbon layers were deposited on silicon microtips that are fabricated as mentioned above. A mixture of semiconductor-grade CH_4 and H_2 was used as a source gas, wherein the fraction of methane was varied between 3.5% and 25%. For all the depositions, the microwave power was 1.0 kW, the chamber pressure was 25 Torr, and the substrate temperature was maintained below 650 °C. It may be noted that heating of the substrates was achieved by the plasma itself without additional heating. A single-color optical pyrometer (Optex VF-850G, Japan) was used to read the temperature from the front surface of the substrate. A negative dc bias (-200 and -300 V) was applied between the graphite substrate holder and grounded chamber wall of the CVD reactor with typical bias for 20, 40, and 60 min.
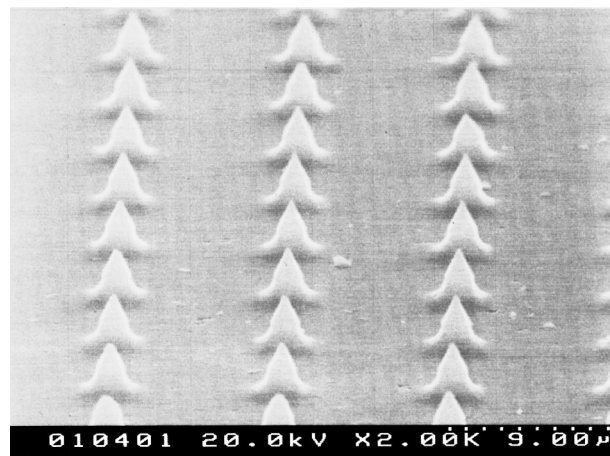

FIG. 2. SEM micrograph of the fabricated BAC Si tip array.

C. Characterization

The morphology and chemical composition of the films were analyzed by SEM and AES. A Perkin Elmer scanning Auger nanoprobe system (SAN 760) using an Ar ion beam with typical beam energy of 3 and 4 keV was used for AES investigation. The typical probe size used was on the order of a few microns, and the system was capable of acquiring depth profile data from an area as small as 500 Å.

Field emission measurements were carried out under high vacuum with a base pressure of about 2×10^{-7} Torr,[9] as shown in Fig. 1. For current–voltage ($I-V$) characteristics, the graphite counter electrode was placed 30 μm above the tip array. A Keithley multimeter was employed for sourcing the voltage and measuring the emission current. In our present work, the emission current (μA) obtained from arrays of 50 $\times$ 50 squares of BAC silicon microtips with 1 μm width is plotted against applied voltage V because the emitting sites are a small fraction of the total film surface of these tips, which makes it difficult to estimate the true current density.

FIG. 3. High-resolution SEM micrograph indicating a tip radius below 55 nm.
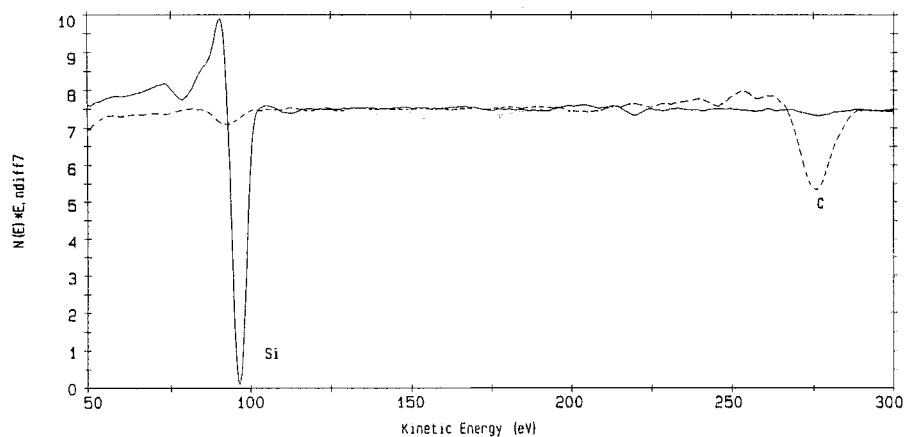
FIG. 4. AES spectra of BAC-treated silicon tip for sample No. 6 (— — —) and untreated silicon microtip (—).

III. RESULTS AND DISCUSSION

A. Morphology and composition of BAC films

A SEM micrograph of the fabricated silicon microtip array is shown in Fig. 2. The carbon layers were uniformly deposited on silicon microtips by the BAC process. Although the apex of the tip is not sharp after coating, the total geometry was not significantly altered from the original shape. The carbon is only selectively deposited on the surface of the sharp silicon tips. This may be attributed to the inherently higher field-enhanced effect near the sharp silicon tips as a strong negative bias is applied on the substrate. The high field near the tip surface leads to a higher concentration of reactive hydrocarbon radicals and carbon ions. The high-resolution SEM micrograph (HRSEM) shown in Fig. 3 indicates that the sharper tip radius can be below 55 nm and shows the smooth surface morphology of deposited carbon layers on the tip.

Figure 4 depicts a typical Auger spectrum of a BAC treated and untreated silicon tip and exhibits major peaks of carbon and silicon, respectively. The peak position around 265 eV in the Auger spectrum indicates that the deposited carbon-based material is an amorphous carbon.[20]

B. Field emission measurements

Field emission characteristics were measured on BAC silicon microtips (arrays of 50×50 squares with 1 μm width) for 3.5%, 15%, and 25% CH_4/H_2 gas ratio with various bias voltages and duration of bias, as listed in Table I. Figures 5(a), 5(b), and 5(c) exhibit the emission current I versus applied voltage V for the field emitter arrays [with the inset being the Fowler–Nordheim (FN) representation of the data], which showed $I-V$ behavior typical of field emission. The good linearity of the FN plots indicated that the electrons were emitted through a field emission mechanism. With maximum accessible applied voltage of 1100 V, emission currents of 80 and 120 μA have been achieved for the films deposited (at dc bias of -200 V for 40 min) with 15% and 25% CH_4/H_2 gas ratio, respectively. An emission current of 40 μA has been achieved for the film grown (at dc bias of -300 V for 40 min) with a 3.5% CH_4/H_2 gas ratio. These values of emission current are comparable with the values of carbon nanostructures,[21] cobalt silicide,[22] and diamond particles[23] on silicon emitters.

A typical dependence of emission on the duration of bias is also observed from Fig. 5(c). The untreated silicon tips are

TABLE I. Deposition conditions and field emission characteristics of bias-assisted carburization of silicon microtips and flat silicon surfaces.

Sample No.	CH_4/H_2 gas ratio (%)	Substrate temperature (°C)	Bias voltage (V)	Duration of bias (min)	Field enhancement factor	Threshold voltage (V)	Emission current (μA at 1100 V)	Current density per array (10^{-3} A/cm^2)	Effective barrier height (eV)
1	3.5	648	-300	40	31	850	36.19	11.46	0.018
2		632		60	32	900	10.30	4.65	0.02
3	15	638		40	31	750	78.16	11.48	0.015
4		639		60	32	1000	4.13	1.36	0.023
5		575		20	32	925	7.59	1.23	0.023
6	25	578	-200	40	32	650	122.70	114.43	0.017
7		590		60	32	950	18.92	17.94	0.027
8[a]		578			...	700	0.92	...	0.50
9[a]	15	638		40	...	700	0.30	...	0.20
10[a]	3.5	648	-300		...	1100	0.01	...	0.30
11[a]	...	...	...	...	28	1050	0.10	0.035	3.76

[a]Samples 8, 9, and 10 represent flat silicon surfaces and sample 11 represents untreated Si tips.
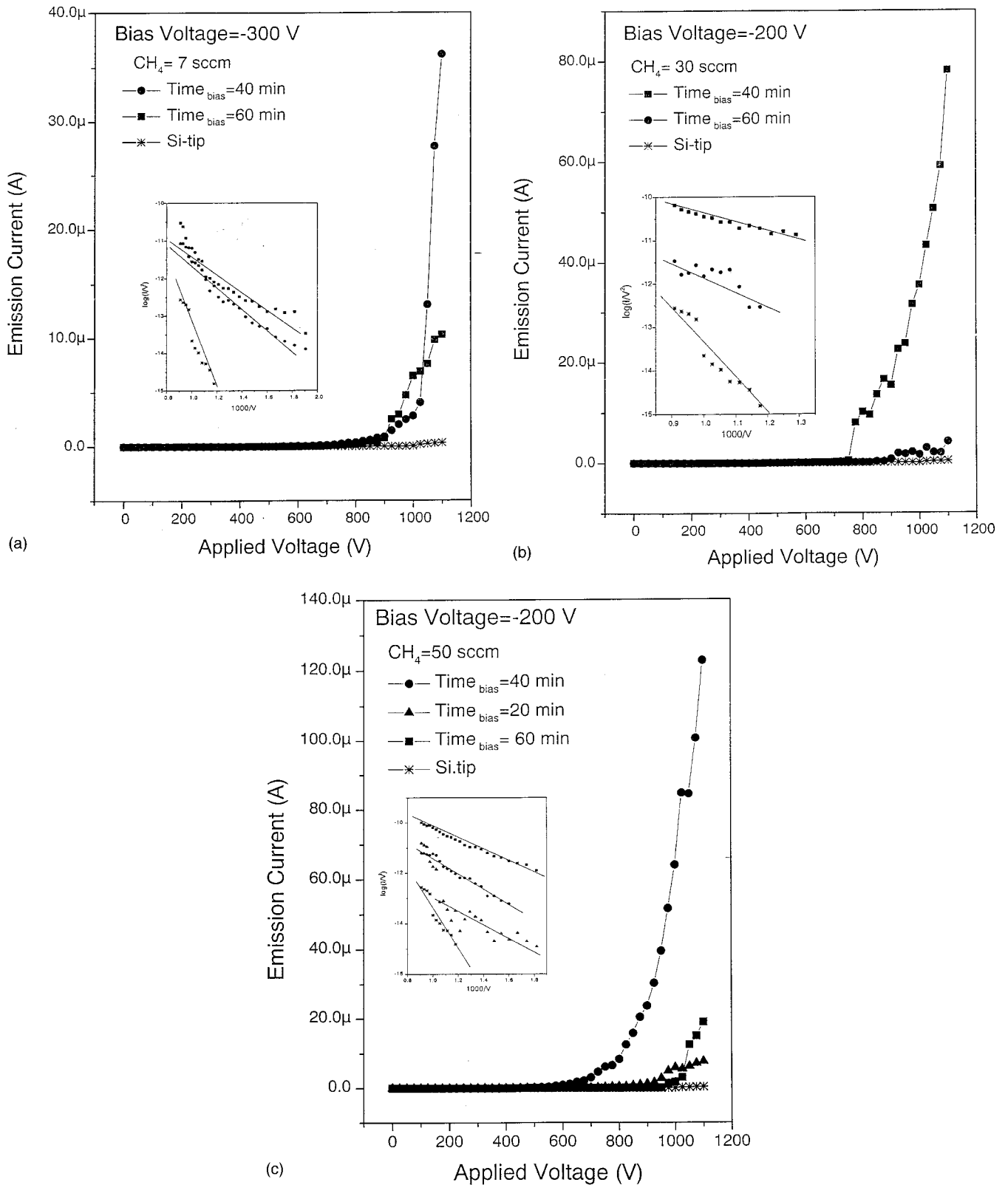
(a)

(b)

(c)

FIG. 5. Field emission $I-V$ curve for BAC silicon microtips using: (a) 3.5% (7 sccm), (b) 15% (30 sccm), and (c) 25% (50 sccm) CH_4/H_2 gas ratio, with the inset being the Fowler–Nordheim representation of the data.
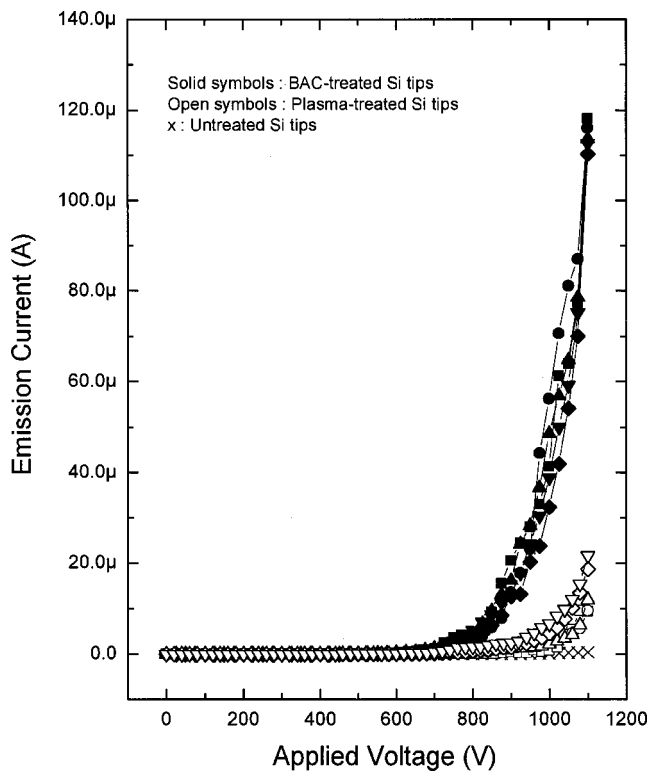
FIG. 6. Field emission $I-V$ curve for five different BAC silicon microtips (solid symbols) and plasma-treated (open symbols) and bare silicon microtips ($\times$). The BAC process was the same as mentioned in Table I for sample 6, whereas plasma-treated silicon microtips were prepared by the above-mentioned condition without adding any CH_4.
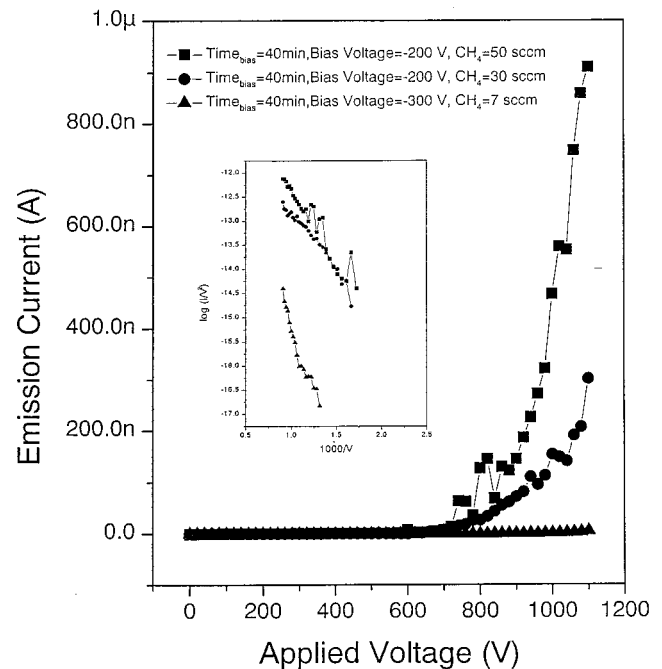

FIG. 7. Field emission $I-V$ curve for three different BAC-treated flat-silicon wafers. The BAC process was the same as mentioned in Table I for samples 1 ($\blacktriangle$), 3 ($\bullet$), and 6 ($\blacksquare$).

not good emitters, but the emission increases with duration of bias, reaching a maximum at 40 min. However, if the tips are overtreated, e.g., 60 min in the present case, the emission current is decreased. This decrease in the emission current is attributed to thicker carbon layers deposited on the silicon microtips (micrograph not shown here), which further leads to blunt silicon tips. It is also observed from Figs. 5(b) and 5(c) that with an increase in CH_4 concentration, the emission current increases and turn-on voltage decreases. Similarly, at higher substrate bias (-300 V), the BAC silicon tips exhibit 40 μA [Fig. 5(a)] emission current. It is reported that an emission current of 57 μA was obtained at an applied voltage of 5 V from a diamond-coated silicon tip,[24,25] whereas a glassy carbon field emitter exhibits an emission current of 50 μA at an applied voltage of 1340 V.[26] The BN coated silicon emitters have demonstrated lower emission threshold voltage and higher emission current of 100 μA.[27] On the other hand, an emission current above 15 μA at an applied voltage of 130 V was obtained from a tungsten-coated silicon emitter.[28] Similarly, diamond-coated Mo emitters have also shown an emission current of 15 μA and are attributed to an electron tunneling through the metal onto the diamond interface.[29,30] The influence of hydrogen plasma annealing, followed by an oxygen plasma treatment, enhances the electron emission of these diamond-coated Mo emitters by formation of Mo_2C carbides at the Mo/diamond interface. Field emission from amorphous diamond-coated Mo tips exhibit an emission cur-

rent of 15 μA.[31] In comparison with bare (untreated) silicon microtips, the emission current of the BAC silicon microtip is enhanced by three orders of magnitude at an applied voltage of 1100 V.

To ascertain the uniformity, reliability, and reproducibility of the field emission, repetitive (twice) field emission measurements on the same samples, as well as on five different samples prepared under a specific treatment have been performed. In the former case, no noticeable change in $I-V$ behavior was detected; in the latter case, the field emission measurements also indicate insignificant variations, as depicted in Fig. 6. Five different BAC-treated silicon microtip samples were prepared in a single batch under the condition mentioned in Table I (for sample No. 6), because this condition gives the highest electron emission current. Similarly, we have also prepared five different plasma-treated silicon tip samples with the above-mentioned condition without adding any CH_4. Figure 6 clearly exhibits not only an enhancement in field emission of BAC-treated silicon tips over plasma-treated bare silicon tips, but also shows a reproducible field emission behavior with the emission current in a range of 112–118 μA at an applied voltage of 1100 V. In addition, the emission current of plasma-treated silicon tips is five times higher than the reported value[32] for bare silicon tips.

To crosscheck the pointed emitters necessary for enhanced field emission, we have selected the three best conditions from Table I (i.e., for samples 1, 3, and 6) and under these conditions, the BAC process was performed on flat Si (100) surfaces. Figure 7 depicts the $I-V$ behavior of these BAC-treated flat-silicon surfaces. With a maximum accessible applied voltage of 1100 V, emission currents of 0.3 and

1 μA have been achieved for the films deposited on flat-silicon wafers (at dc bias of -200 V for 40 min) with 15% and 25% CH_4/H_2 gas ratios, respectively. An emission current of 0.025 μA has been achieved for the film grown on flat-silicon wafers (at dc bias of -300 V for 40 min) with 3.5% CH_4/H_2 gas ratio. Thus, all these values are much lower in comparison with BAC-treated silicon microtips.

The field emission characteristics of the BAC-treated silicon microtips are further analyzed by the FN plot, a plot of log I/V^2 vs $1/V$, as in the inset of Fig. 5. The plot yields a straight line according to the FN equation[33]

$$J = \frac{A}{\phi}\left(\frac{\beta V}{d}\right)^2 \exp\left(\frac{-B\phi^{3/2}}{\beta(V/d)}\right), \tag{1}$$

where A and B are constants, current density J is in A/m^2, voltage V is in V, anode-to-cathode distance d is in m, ϕ is the effective barrier height for electrons in eV, and $\beta = h/r$ is the field enhancement factor[34] that depends on the emitter geometry, where h represents the tip height and r is the radius of the tip emitter. Both these values are calculated from the HRSEM image of the microtips. The current emitted by field emitter arrays is given by $I = nA'J$, where A' is emission area per tip and n is the number of emitters. Making the appropriate substitutions in Eq. (1), one gets Eq. (2):

$$I = \frac{nA'A}{\phi}\left(\frac{\beta V}{d}\right)^2 \exp\left(\frac{-B\phi^{3/2}}{\beta(V/d)}\right). \tag{2}$$

Alternatively, Eq. (2) can be expressed as

$$\log \frac{I}{V^2} = -\log \frac{\phi}{nA'A}\left(\frac{d}{\beta}\right)^2 - \left(\frac{B\phi^{3/2}d}{\beta}\right)\frac{1}{V}. \tag{3}$$

Consequently, a plot of log (I/V^2) vs I/V yields a straight line with slope $B\phi^{3/2}d/\beta$. The effective barrier heights, field enhancement factors, and current density for each array were determined from the slope of FN plots, HRSEM images, and substitutions of appropriate values in Eq. (3), respectively, for various BAC silicon tips and are presented in Table I. The values of the emission barrier and field enhancement factor were found to be in the range of 0.015–0.027 eV and 31.06–31.62, respectively, as listed in Table I. For plasma-treated Si tips, the effective barrier height and field enhancement factor were estimated to be 0.042 eV and 30.86, respectively. Notwithstanding, it is informative to note that the value of the emission barrier of plasma-treated Si tips is much lower than reported for elemental silicon (3.8 eV),[35] whereas for untreated Si tips, the value of the emission barrier is consistent with that of the value reported. The values of effective barrier height of BAC silicon microtips are also low compared to diamond (0.37 eV),[36] tungsten (0.5 eV),[37,38] and gold (1.7 eV)[37,38] surfaces. However, the values of effective barrier height of BAC silicon microtips are consistent with the values reported for graphite-type carbon coatings on silicon (0.02 eV),[39] nitrogen-containing hydrogenated amorphous carbon films on n^{++}-Si (0.03–0.05 eV),[40] and polycrystalline diamond films on silicon (0.08 eV).[34] The values of effective barrier height of BAC treated flat silicon surfaces were found to be in the range of 0.2–0.5 (listed in Table I)
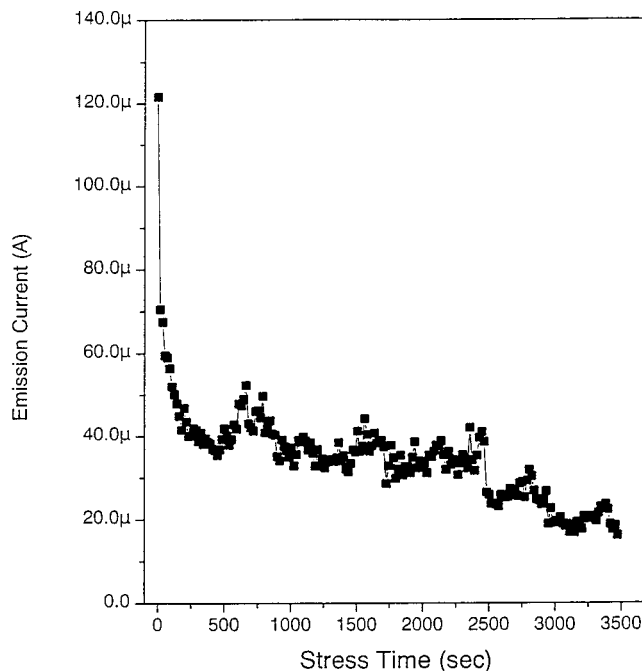

FIG. 8. Emission stability at a constant voltage of 1100 V for sample 6 (as listed in Table I).

and are higher than BAC-treated Si tips. The field emission characteristics of BAC-treated silicon tips are similar to emission properties of cobalt silicide,[22] diamond particles,[23] and tungsten-coated[28] silicon emitters. Thus, it is evident from the above observation that the effective barrier height deduced from the field emission measurements does not represent the work function of the materials. The values of the field enhancement factor are approximately the same for all BAC silicon microtips. On the other hand, the field enhancement factor for single-wall carbon nanotubes[13] is higher than 1000, but there is a significant uncertainty in the origin of the low field emission from carbon nanotubes. The emission site density is also very low, indicating that only a few nanotubes emit. The scanning field emission from patterned carbon nanotube films reveals that the carbon nanotubes with a lower length-to-diameter ratio are not good emitters, and the field emission is governed by the relative height of nanotubes as compared to the intertube distance.[41] It is also observed from Table I that with an increase in CH_4 concentration, the values of effective barrier height remain almost constant. Thus, the low energy barrier of BAC silicon tips and/or large field enhancement factor enhances the electron emission of BAC silicon microtips.

Figure 8 shows the evolution of emission current with operating time at a constant voltage of 1100 V for sample 6 (listed in Table I). A sharp decay of the emission current during the initial 250 s, from 120 μA to one-third of the initial value, is observed. The emission current then decreases monotonically with time. The reduction in emission current is caused not only by the damage to the surface of the silicon tip arrays [as seen from Fig. 9(a)], but also by the damage to the BAC silicon microtips as the tip became blunt
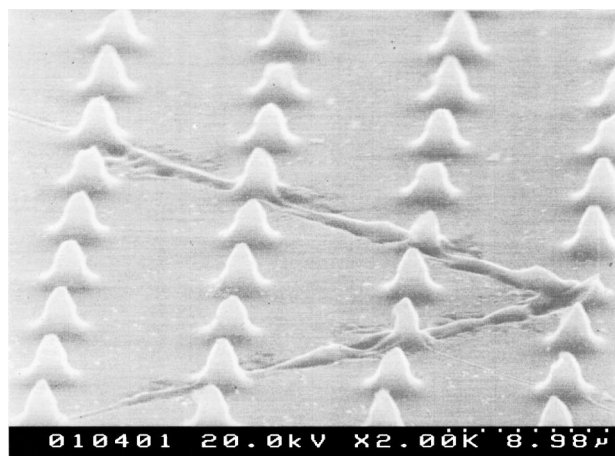
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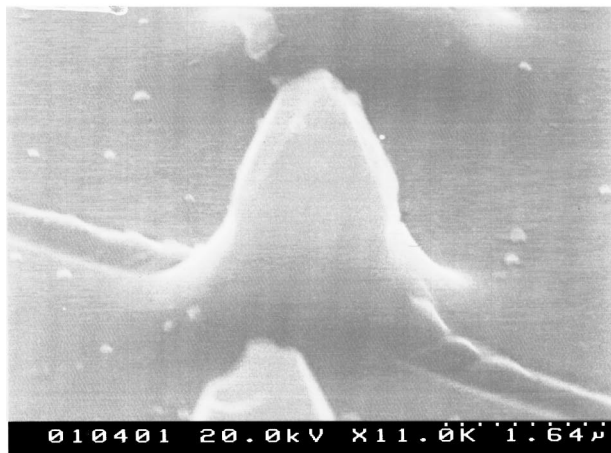
(b)

FIG. 9. (a) High-resolution surface morphology of Si tip array and (b) surface morphology of BAC silicon microtip after a high applied voltage of 1100 V for 1 h for sample 6 (as listed in Table I).

[as observed in Fig. 9(b)]. Thus, comparing the emission behavior of BAC silicon tips with the untreated one, we deduce that the emission of the BAC silicon tip array not only depends on selectivity and thickness of the BAC layer deposited on silicon tips, but also on surface morphology and geometry of these tips.

IV. CONCLUSIONS

We have investigated the field emission characteristics of silicon microtips array covered with ultrathin carbon layers deposited on ultrasharp silicon microtips by BAC using MWPCVD. The BAC process was performed at low pressure and low deposition temperature. The BAC layer was selectively deposited on the surface of the silicon tip with a substrate bias of -200 V. The outstanding features of our present studies are: (1) The field emission is enhanced by about three orders of magnitude in BAC silicon microtips over untreated silicon microtips. (2) There is a rapid decrease in the field-emission threshold voltage with an increase in CH_4 concentration in the plasma. (3) The lowest emission

threshold voltage and higher emission current were observed for the BAC silicon microtips grown with 50 sccm CH_4 concentration at a dc bias of -200 V for 40 min. The higher emission current of the BAC silicon microtip (at -200 V) is attributed to the lower values of the effective barrier height of these tips as compared to pure and plasma-treated silicon tips. Furthermore, the BAC silicon microtip has good emission stability at a constant applied voltage of 1100 V. The high emission current, low temperature, and large-area growth capability of the BAC silicon tip emitter and a simple coating by a novel method are promising features of our investigation. Because there is great interest and need for improved emitter performance in terms of reliability and low turn-on voltage, the BAC process on silicon microtips reported here offers great potential in this area.

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